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PROCESSING PROCEDURE AND FLAVOR STABILITY IN SOYBEAN OIL[1]

BY H. J. LIPS,[2] N. H. GRACE,[2] AND J. A. ZIEGLER[3]

ABSTRACT

Chemical and physical measurements and small scale taste panel tests on laboratory refined American and Canadian solvent extracted soybean oils indicated that their quality was generally similar. Flavor stability was not improved by deodorization in the presence of citric acid, increased deodorization time, treatment with activated magnesia, preliminary treatment with concentrated hydrochloric acid, or by refining in miscella with or without subsequent addition of butylated hydroxyanisole.

INTRODUCTION

Methods for delaying the onset and decreasing the extent of flavor reversion in soybean oil have been under active study for some time in the United States (7, pp. 812-831). Increasing domestic production of soybeans and larger imports of beans and oil have emphasized the importance of this problem in Canada. Flavor stability studies on soybean oil have therefore been initiated, including a comparison of United States and Canadian extracted oils, the effects of various modifications in conventional refining procedures, and a comparison of miscella and conventional refining.

MATERIALS AND METHODS

A. *Experiments with Oils Refined by Conventional Procedures*

Commercial, crude, solvent extracted soybean oils prepared by American and Canadian continuous methods and by a Canadian semicontinuous method were given the conventional laboratory Treatment 1 shown below. Treatments 2 to 5 were applied to the first two oils only, to study the effects of variations in conventional refining. Treatments were performed independently on two lots of each oil.

[1] *Manuscript received October, 1951.*
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Treatment		References
1	Standard. Alkali refined at 60°C. with 20°Bé. sodium hydroxide, bleached with 2% Superfiltrol for 20 min. under nitrogen, and deodorized one hour at 240°C.	(5), (6)
2	Same as 1, but deodorized in the presence of 0.02% citric acid, anhydrous basis.	(1)
3	Same as 1, but deodorized four hours.	(9)
4	Same as 1, but deodorization preceded by treatment with 0.5% of activated magnesia for three hours at 50°C.	(8)
5	Same as 1, but crude oil given a preliminary treatment with 1% of concentrated hydrochloric acid, and a heat treatment after alkali refining.	(3)

Subjective appraisals of the intensity of undesirable odor in the oils themselves, and of undesirable odor or flavor of doughnuts fried in the oils, were made independently by a 24-member panel. Samples were appraised in randomized sets of 6 or 7, and scored on the customary 6 point scale (6), with "0" representing the ideal condition and + 1 to + 5 representing increasing degrees of objectionable odor or flavor. For odor tests, the oils were heated at 200°C. for 15 min., and then cooled and served at room temperature. Doughnuts were served warm. Commercial cottonseed salad oil was used without further processing as a reference material in all taste panel tests. Statistical treatment of panel scores emphasized comparison between treatments on any one oil, and gave a limited comparison among the three soybean oils for Treatment 1 only.

The usual chemical tests (2, 6) were applied to the fresh and used oils. For determination of stability to oxidative deterioration, the oils were stored at 60°C. and examined at convenient intervals for peroxide oxygen development.

B. *Experiments with Oils Refined in Miscella*

Three lots of commercial soybean miscella were each divided into equal parts. One half of each lot, on evaporation of the hexane solvent, yielded oil which was refined by conventional procedures. The remaining portion of each lot was refined in miscella prior to removal of the solvent. Miscella containing approximately 56% oil by weight was bleached with activated earth* (3% by weight of oil) for three minutes at 55°C. After removal of the clay by filtration, the bleached miscella was alkali refined at 50°C. with a solution of sodium hydroxide containing sodium chloride. This use of "caustic brine" led to a more rapid separation of the soap and fat phases.

The six samples of soybean oil were deodorized for one hour at 240°C. in the presence of 0.02% citric acid, which was added to the deodorizer flasks as a solution in a few milliliters of purified absolute ethanol. For taste panel and storage tests each deodorized sample was halved, and 0.02% butylated hydroxyanisole (4) was added to half of each sample. This antioxidant was

**Tonsil AC from Western Germany.*

dissolved by warming in a few milliliters of oil, which was then mixed with the remaining oil. Taste panel and objective tests were carried out as described above.

RESULTS

A. *Oils Refined by Conventional Procedure*

Objective measurements showed little variation among the original crude soybean oils. The iodine value, refractive index, free fatty acid content, viscosity, light transmission, and polyunsaturated acid composition of the crude soybean oils were generally similar (Table I). Comparative values for some measurements are shown for the cottonseed oil.

TABLE I

CHARACTERISTICS OF EXPERIMENTAL OILS

Characteristics	Cottonseed salad oil	Crude soybean oils		
		American continuous process	Canadian continuous process	Canadian semicontinuous process
Iodine value	109.8	122.6	123.2	122.9
Refractive index at 130°F.	1.4593	1.4610	1.4618	1.4615
F.F.A., as % oleic	0.0	0.9	1.0	0.9
Viscosity, centistokes at 130°F.	21.0	20.1	20.1	19.6
Light transmission, % at:				
440 mμ	57	3	3	3
520 mμ	89	16	8	12
660 mμ	97	65	50	65
% Linoleic acid	—	51.0	45.6	49.4
% Linolenic acid	—	6.8	6.4	6.5

Since the two soybean oils refined by Treatments 1 to 5 showed only minor differences in iodine value, refractive index, and viscosity, the data are not recorded. Free fatty acid content, as per cent oleic acid, did not exceed 0.2% for any of the refined oils at any stage of processing and use. The ranges of values for light transmission, smoke point, and peroxide value are shown in Table II for soybean oils freshly deodorized, after use in the rapid heat test, and after use for deep fat frying of doughnuts. Values for cottonseed oil are shown for comparison. The lowest values obtained for light transmission of soybean oils resulted from Treatment 4, which produced darker colored oils of a greenish shade. The other treatments gave oils of normal color which did not differ from each other in appearance. Smoke points ranged from 410° to 455°F. Peroxide values were low, with the highest values from Treatment 4, corresponding to the more objectionable flavor and odor shown for this treatment. Storage results (not shown) indicated a slight retardation of peroxide formation by Treatment 2 (deodorization with citric acid), with no other differences according to source or treatment of soybean oil.

All oils heated to 200°C., including the cottonseed oil, were judged to have some undesirable odor, with average odor scores ranging around +1 (Table

TABLE II

RANGE OF CHARACTERISTICS OF REFINED EXPERIMENTAL OILS

Oils	Characteristics				
	Light transmission, % at:			Smoke point, °F.	Peroxide value, ml. 0.002 *N* thio*** per gm.
	440 mμ	520 mμ	660 mμ		
Cottonseed salad					
As purchased	57	89	97	445	1.0
Heated	60-61	92-93	99-100	440-445	. . .
Used for frying	54-55	89-90	96-97	440-450	1.9-2.0
American continuous soybean*					
Deodorized	65-76	90-95	86-100	430-455	. . .
Heated	61-69	91-96	90-100	415-450	. . .
Used for frying	49-65	87-94	86-100	415-445	1.4-4.4
Canadian continuous soybean*					
Deodorized	54-72	84-94	78-99	430-440	. . .
Heated	49-66	86-95	80-100	410-445	. . .
Used for frying	47-61	83-94	79-100	415-445	1.4-4.4
Canadian semicontinuous soybean**					
Deodorized	67-70	92-94	97-99	435-445	. . .
Heated	70-71	96	100	420-430	. . .
Used for frying	64-65	93-94	100	430	1.6-1.7

**The ranges shown cover all refining treatments (1-5).*
***The ranges shown are for Treatment 1 only.*
****Sodium thiosulphate.*

TABLE III

AVERAGE PANEL RATINGS FOR TEST OILS AND FOR DOUGHNUTS FRIED IN TEST OILS

Panel appraisals	Soybean oils	Soybean oil treatments					Cotton-seed oil	Necessary difference, 5% level
		1	2	3	4	5		
Intensity of undesirable odor of heated oils	American continuous process							
	First lot	1.2	1.5	1.1	1.9	1.4	1.5	0.7
	Second lot	1.2	1.4	1.2	2.3	1.5	1.3	0.7
	Canadian continuous process							
	First lot	1.0	1.1	1.8	2.1	2.0	1.4	0.7
	Second lot	1.2	1.1	1.1	2.5	1.2	1.3	0.7
Intensity of undesirable odor of doughnuts	American continuous process							
	First lot	0.8	1.3	0.9	1.2	0.8	1.1	0.6
	Second lot	0.5	0.8	1.1	1.6	1.8	1.1	0.7
	Canadian continuous process							
	First lot	0.5	0.6	0.7	2.3	2.3	0.8	0.7
	Second lot	1.0	1.0	1.0	2.7	1.0	0.8	0.7
Intensity of undesirable flavor of doughnuts	American continuous process							
	First lot	0.7	1.5	0.8	1.2	0.8	0.7	0.6
	Second lot	0.9	0.5	1.0	1.6	2.2	0.7	0.7
	Canadian continuous process							
	First lot	0.9	0.7	0.8	2.6	2.8	0.8	0.7
	Second lot	0.5	0.8	0.8	3.2	0.8	0.7	0.7

III). Standard refining (Treatment 1) gave oils with the least undesirable odor (lowest score). None of the experimental treatments improved the odor of the American or Canadian continuous process soybean oils, and Treatment 4 with activated magnesia appeared to increase objectionable odor.

For doughnut frying the standard processed soybean oil (Treatment 1) again gave the best results (Table III). Refining with activated magnesia (Treatment 4) or hydrochloric acid (Treatment 5) had an adverse effect (increased scores).

Taste panel trials of soybean oil prepared by the Canadian semicontinuous process and refined according to the standard method (Treatment 1) showed that this oil did not differ significantly from the similarly refined American and Canadian continuous process soybean oils. Average odor and flavor scores for oils and doughnuts were all about + 1.

B. *Oils Refined in Miscella*

No important differences in characteristics between standard and miscella refined soybean oils were noted at any stage of processing or use. Some characteristic data are shown in Table IV, with comparative values for cottonseed oil. Storage tests (data not given) indicated that butylated hy-

TABLE IV

COMPARATIVE CHARACTERISTICS OF SOYBEAN OIL REFINED BY STANDARD PROCESSES AND REFINED IN MISCELLA

Oils	Characteristics					
	Light transmission, % at:			Peroxide value, ml. 0.002 N thio per gm.	F.F.A., as % oleic	Smoke point, °F.
	440 mμ	520 mμ	660 mμ			
Original						
Standard process soybean	64	94	100	—	—	—
Miscella process soybean	68	95	100	—	—	—
Cottonseed salad	64	92	98	—	—	—
Deodorized						
Standard process	72	95	99	—	—	420
Miscella process	65	92	99	—	—	390
Standard + antioxidant	86	97	99	—	—	415
Miscella + antioxidant	64	83	94	—	—	385
Heated						
Standard process	85	97	100	0	0.1	420
Miscella process	69	89	98	0	0.4	385
Standard + antioxidant	70	94	99	2.6	0.1	415
Miscella + antioxidant	62	90	97	1.7	0.3	380
Cottonseed salad	63	91	98	5.5	0.0	445
Used for frying						
Standard process	69	94	99	1.3	0.1	420
Miscella process	62	91	97	1.5	0.3	380
Standard + antioxidant	68	94	99	1.1	0.1	415
Miscella + antioxidant	61	90	98	1.5	0.3	385
Cottonseed salad	61	92	99	2.0	0.0	445

TABLE V

AVERAGE PANEL RATINGS FOR TEST OILS AND FOR DOUGHNUTS FRIED IN TEST OILS
(Miscella experiment)

Panel appraisals	Soybean oils	Soybean oil treatments			Cottonseed oil
		Without antioxidant	With antioxidant	Average	
Intensity of undesirable odor of heated oils	First lot				
	Standard refining	1.4	1.3	1.4	0.9
	Refined in miscella	1.6	1.4	1.5	
	Average	1.5	1.4		
	Second lot				
	Standard refining	1.0	1.3	1.2	1.2
	Refined in miscella	1.7	2.0	1.9	
	Average	1.4	1.7		
	Third lot				
	Standard refining	0.9	1.0	1.0	0.8
	Refined in miscella	1.8	2.0	1.9	
	Average	1.4	1.5		
Intensity of undesirable odor of doughnuts	First lot				
	Standard refining	0.5	0.3	0.4	0.6
	Refined in miscella	0.3	0.3	0.3	
	Average	0.4	0.3		
	Second lot				
	Standard refining	0.6	0.8	0.7	0.2
	Refined in miscella	0.5	0.3	0.4	
	Average	0.6	0.6		
	Third lot				
	Standard refining	0.4	0.2	0.3	0.3
	Refined in miscella	0.6	0.3	0.5	
	Average	0.5	0.3		
Intensity of undesirable flavor of doughnuts	First lot				
	Standard refining	0.5	0.2	0.4	0.3
	Refined in miscella	0.3	0.5	0.4	
	Average	0.4	0.4		
	Second lot				
	Standard refining	1.3	1.0	1.2	0.3
	Refined in miscella	0.7	0.8	0.8	
	Average	1.0	0.9		
	Third lot				
	Standard refining	0.4	0.5	0.5	0.3
	Refined in miscella	0.7	0.5	0.6	
	Average	0.6	0.5		

	Necessary difference, 5% level:	
	Between single items, same lot	Between averages of two items, same lot
Odor of oils	0.5	0.4
Odor of doughnuts	0.6	0.4
Flavor of doughnuts	0.7	0.5

droxyanisole had about the same slight antioxidant effect for standard and miscella process soybean oils.

The taste panel considered that all of the heated oils, including the comparison samples of cottonseed oil, had a slight to moderate intensity of undesirable flavor (Table V). In the second and third lots of soybean oil, that refined in miscella was, on the average, slightly more objectionable in odor than that refined by the standard method. The panel did not discriminate significantly among the odors and flavors of doughnuts fried in any of the test oils. Soybean oils of the second lot were considered to have imparted more undesirable flavor to the test doughnuts than did the cottonseed oil with, however, no significant difference between "standard" and "miscella" process oils in this respect. Addition of antioxidant (butylated hydroxyanisole) had no demonstrable effect on odor or flavor scores.

During frying of doughnuts in miscella refined oil a gelatinous brown precipitate was formed. This effect was particularly marked with the second lot of oils tested. The formation of a precipitate, and a related cloudiness of miscella refined oil after deodorization, indicated that some material in this oil, not present in oil refined by the standard process, was rendered insoluble by hydration. The contaminant was later found to be a small amount of break matter containing a trace of soap and could be eliminated from the miscella refined oils by two water washes.

DISCUSSION

The results indicate no improvement in flavor stability of soybean oil by the use of various modifications in processing procedure. Some of the recommended changes may also cause other difficulties, e.g., increased color with Treatment 4.

Various reasons may be advanced for the apparent lack of improvement. The difficulty in reproducing processing conditions is reflected in the unexpectedly wide divergencies in a few of the taste panel scores for duplicate lots of oil (Table III). Furthermore, in laboratory experiments largely done in glass, procedures designed to take care of heavy metal contamination (i.e., as with citric acid) may be superfluous. Finally, the rather broad recommendations given in some of the patents may not have been applied in the most effective range of temperature, time, etc. However, as recently stated (7, p. 831), "it would appear . . . that no process has yet been developed which is both practical and significantly effective in retarding or preventing the development of off-flavors in (soybean) oil".

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